



# Formation and efficacy of TiO<sub>2</sub>/AC composites prepared under microwave irradiation in the photoinduced transformation of the 2-propanol VOC pollutant in air



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## ABSTRACT

This article reports on the preparation and characterization (SEM, SEM-EDX, XRD, diffuse reflectance spectroscopy, and BET surface area) of TiO<sub>2</sub> particles supported on activated carbon (AC) particulates using a titanium oxysulphate precursor and subjecting the aqueous dispersion to microwave (MW) heating and to a more traditional heating method with an oil bath. The TiO<sub>2</sub>/AC composites were subsequently tested for their photoactivity through an examination of the transformation of a volatile organic pollutant (VOC) in air: iso-propanol. Under MW irradiation at 70 °C the synthesis resulted in the formation of a thin coating about the AC support, while TiO<sub>2</sub> particles formed at higher temperatures; the average particle size of TiO<sub>2</sub> tended to decrease with increase in reaction temperature from 426 nm at 80 °C to 243 nm at 180 °C. The accelerated heating of the AC-dispersed solution above 80 °C was confirmed by determining the dielectric loss ( $\epsilon''$ ) of the dispersion at various temperatures at the microwave frequency of 2.45 GHz. Subjecting the dispersion to oil-bath heating only led to formation of a thin film about the AC particulates. In the absence of the AC support TiO<sub>2</sub> particle sizes averaged ca. 460 nm for the MW method, while they averaged around 682 nm with the oil-bath method. The BET specific surface area of the TiO<sub>2</sub>/AC composites was significantly greater for the MW heating method (ca. 990 m<sup>2</sup> g<sup>-1</sup> versus 848 m<sup>2</sup> g<sup>-1</sup> for the oil-bath method). Both UV–vis spectroscopy (estimated band-gap energy of TiO<sub>2</sub>/AC composites was 3.3 eV) and XRD spectra confirmed the anatase nature of the TiO<sub>2</sub> specimens. The MW-produced TiO<sub>2</sub>/AC particulates proved to be nearly six-fold more photoactive in the photoinduced degradation of the VOC pollutant than those produced by the oil-bath method. A possible growth mechanism of the TiO<sub>2</sub>/AC composites is proposed.

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## 1. Introduction

Advanced oxidation processes (AOPs) have proven through the years to be effective in the photooxidative disposal of various volatile pollutant materials both in the gas phase and in aqueous media [1]. The most widely adopted AOPs include photodegradation in the presence of TiO<sub>2</sub>, the Fenton and photo-Fenton processes, together with ultrasonication and ozonation (O<sub>3</sub>). AOPs rely on the generation of reactive free radicals, especially the hydroxyl and hydroperoxyl radicals ( $\cdot\text{OH}$ ,  $\text{HOO}\cdot$ ), and the superoxide radical anion ( $\text{O}_2^{\cdot-}$ ). Removal of environmental pollutants through semiconductor photocatalysis has attracted extensive interest over the last few decades. Among various semiconductors,

TiO<sub>2</sub> has been known as the leading photocatalyst due to its good photoactivity, high chemical stability, low cost, and nontoxicity [2].

Significant progress has been made in recent years in immobilizing titanium dioxide particles on such supporting materials as glass, silica beads, polymer and zeolite in heterogeneous photocatalysis [3], as reusing dispersed TiO<sub>2</sub> nanoparticles causes undue problems of filtration. Moreover, we must recognize that nanoparticles have an important consequence on human health and on ecological systems [4]. Accordingly, it is important to fix TiO<sub>2</sub> particles for possible recycling of photocatalytic events. Immobilization of TiO<sub>2</sub> on a support through loading these nanoparticles on activated carbon (AC) has drawn great attention owing to the high adsorption capability of activated carbon that can help to enrich an organic substrate close to the catalyst, thereby promoting pollutant transfer from the support onto the photocatalyst TiO<sub>2</sub> and increasing photocatalytic efficiency. The synergistic effect of adsorption by AC and TiO<sub>2</sub> particulates can have beneficial consequences in the photo-induced transformation of several types of organic

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pollutants such as the defluorination of pentafluorobenzoic acid [5] and the photodegradation of a dye [6] in aqueous media, and the photodecomposition of the volatile organic compound (VOC) iso-propanol in air [7] in the presence of dispersed  $\text{TiO}_2/\text{AC}$  particles.

Preparation of the composite  $\text{TiO}_2/\text{AC}$  particles has been achieved through mechanical mixing of  $\text{TiO}_2$  and AC particles [6], and through dipping AC particles in titanium (IV) *iso*-propoxide solution [8]. In the present study, the  $\text{TiO}_2/\text{AC}$  composite photocatalyst particles were prepared using a microwave hydrothermal synthesis method using the more stable titanium oxysulphate as the  $\text{TiO}_2$  source in water compared to titanium (IV) *iso*-propoxide. The growth mechanism of  $\text{TiO}_2$  on the AC surface was examined by the microwaves' selective heating; the photoactivity of the  $\text{TiO}_2/\text{AC}$  composites was evaluated using the decomposition of *iso*-propanol.

## 2. Experimental

### 2.1. Preparation of $\text{TiO}_2/\text{AC}$ particles

An aqueous titanium oxysulphate solution (0.125 M; 20 mL) and activated carbon (AC; 1 g; diameter: 0.65 nm) were introduced into an Anton Paar high-pressure Pyrex cylindrical reactor (30 mL), following which the reactor was subjected to microwave irradiation under stirring conditions (400 rpm) using an Anton Paar Monowave 300 microwave apparatus. Determination of the temperature distribution in a reactor poses a frequent problem when using microwave heating [9]. Accordingly, the temperature distribution in the sample solution was measured using both a ruby fiber optic sensor located at the center of the solution and a radiation thermometer near the external wall of the reactor. The difference in temperature of the solution at the two locations was less than 2 °C, indicating a nearly uniform temperature distribution throughout the suspension. Soon after the microwave heating step, the sample of  $\text{TiO}_2/\text{AC}$  particles was filtered and washed repeatedly with methanol and water, and then dried at 500 °C overnight in an electric furnace.

The reaction temperature was controlled by a proportional-integral-differential control system, which was attained in 45–48 s; after reaching the desired reaction temperature, the suspension was kept at this temperature for 5 min. The sample was subsequently cooled rapidly with an intense air flow from an air compressor. Fourteen reaction temperatures were used in the range 70–200 °C at 10 °C steps. For comparison, we also used conventional heating with an oil bath in the synthesis of  $\text{TiO}_2/\text{AC}$  particles under otherwise identical temperature conditions achieved by soaking the cylindrical reactor in the oil bath pre-heated to 170–190 °C.

Color changes occurring during the synthesis of  $\text{TiO}_2$  particles accompanying heating were observed with a standard optional CCD camera attached to the Anton Paar Monowave 300 microwave apparatus. Adsorption of  $\text{TiO}_2$  particles on the surface of activated carbon under microwave heating and particle sizes of the particulates were monitored by scanning electron microscopy (SEM). The UV–vis absorption spectra were analyzed with a JASCO V-660 double-beam spectrophotometer equipped with a JASCO ISV-722 integrating sphere; a WWBG-773 program was used to estimate the band gap energy. X-ray patterns of the  $\text{TiO}_2/\text{AC}$  composite particles and of the dispersed  $\text{TiO}_2$  in solution produced by the microwave method (90 °C) were recorded with a Philips X-ray diffractometer (X'pert PRO). The Brunauer–Emmett–Teller (BET) specific surface area of the synthesized  $\text{TiO}_2/\text{AC}$  composites was measured using the Quantachrome Autosorb 3B analyzer.

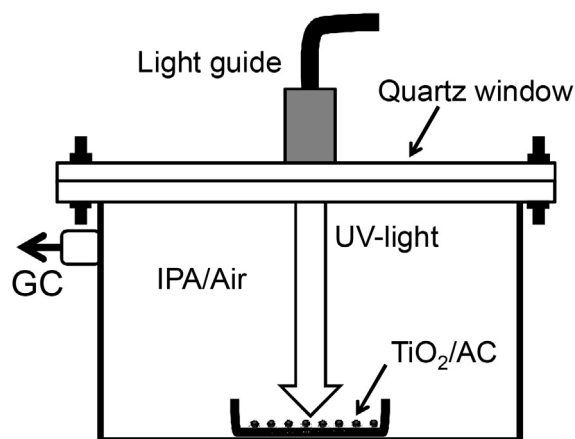


Fig. 1. Schematic of the experimental setup used in the photodecomposition of IPA on  $\text{TiO}_2/\text{AC}$  particles in air by UV irradiation.

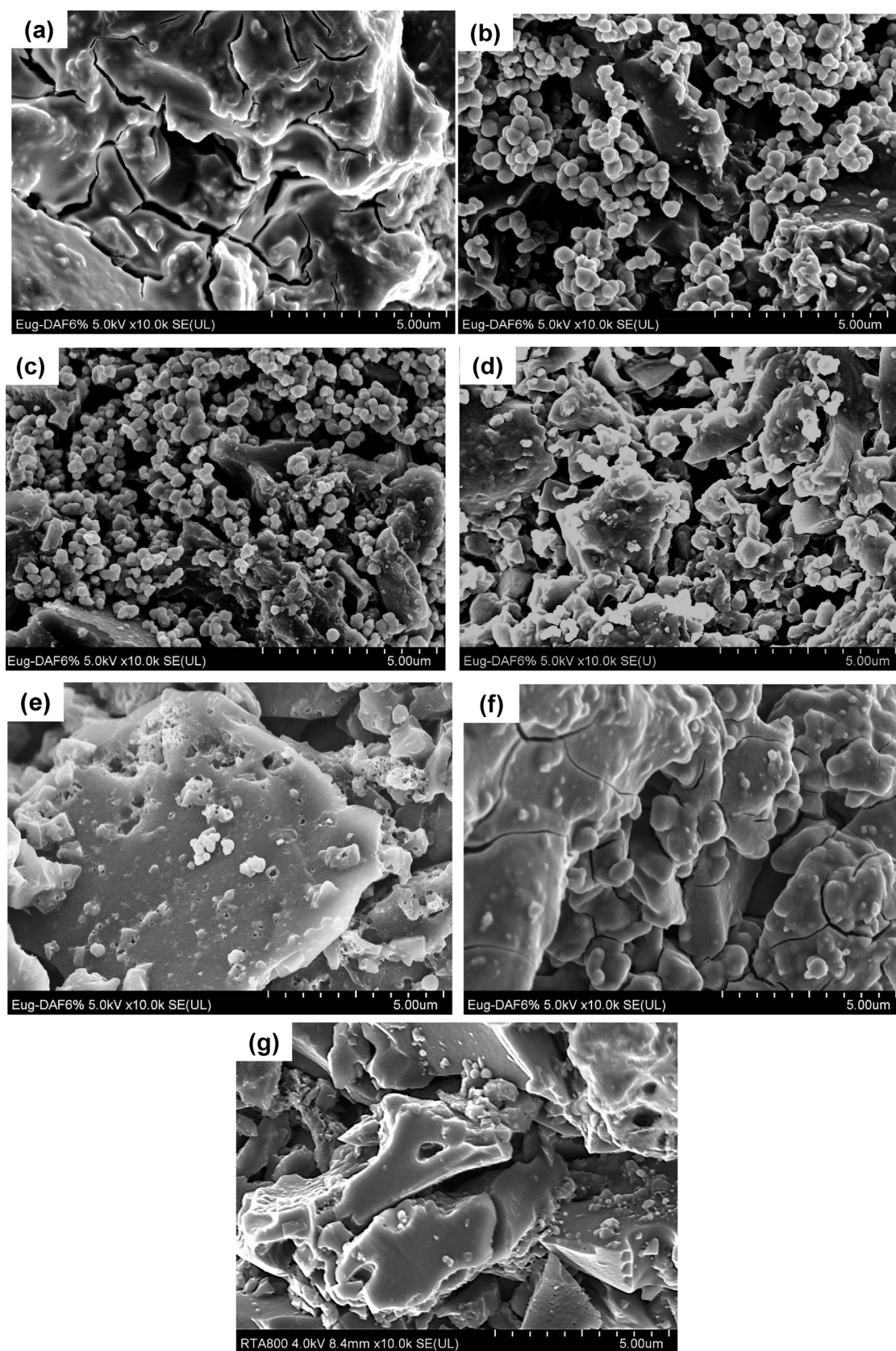
### 2.2. Photoactivity of $\text{TiO}_2/\text{AC}$ using the UV-driven and microwave-assisted photodegradation of *iso*-propanol

Evaluation of the photoactivity of  $\text{TiO}_2/\text{AC}$  particulates was made by the photodecomposition of *iso*-propanol (IPA; Wako Pure Chemical Industries, Ltd.) as a volatile organic pollutant in air. The Pyrex glass batch reactor (internal diameter: 100 mm; internal height: 60 mm) with a quartz lid is illustrated in Fig. 1.  $\text{TiO}_2/\text{AC}$  particles were placed in a petri dish (100 mg), followed by injecting the IPA (2000 ppm) in the closed reactor under dark conditions. The system was allowed to stand in the dark for 40 min so as to achieve equilibrium adsorption of IPA onto the  $\text{TiO}_2/\text{AC}$  particulates, after which the system was UV irradiated using a San-Ei Supercure-203S high pressure Hg-lamp (200 W) through a light guide positioned on top of the quartz window. The temporal decrease of IPA concentration was periodically measured with a Shimadzu gas chromatograph (GC-2014; GL Science, Sorbitol column).

## 3. Results and discussion

### 3.1. Preparation of $\text{TiO}_2/\text{AC}$ particles

The initial transparent solution became cloudy at 70 °C, turning to a white dispersion of  $\text{TiO}_2$  particles generated at 80 °C; at 90 °C the white color of the suspension was enhanced even further. The  $\text{TiO}_2$  particles formed a thin film coating on the activated carbon surface at the heating temperature of 70 °C as illustrated in Fig. 2a and confirmed by the SEM-EDX technique (Fig. 3), whereas  $\text{TiO}_2$  particles formed on the AC surface at 80 °C (Fig. 2b). Selecting some 50 particles of  $\text{TiO}_2$  chosen at random from the SEM photographs revealed that the average particle size of  $\text{TiO}_2$  on the AC surface was 426 nm at the reaction temperature at 80 °C, while at the reaction temperature of 90 °C the average particle diameter decreased slightly to 415 nm (see Fig. 2c). When the reaction temperature increased to 120 °C (note that since the reactor used in this experiment is of a closed type, temperatures greater than 100 °C can be achieved changing the microwave input power)  $\text{TiO}_2$  particles were seen to be adsorbed on the AC surface as evidenced in the SEM image of Fig. 2d, which shows small (357 nm) and larger size intermingled particles on the AC surface. Fig. 4 displays a plot of the monotonic decrease of  $\text{TiO}_2$  particle size on the AC surface at various reaction temperatures. Clearly, the  $\text{TiO}_2/\text{AC}$  composite particles from the synthesis at the higher temperatures tended to have lower specific surface areas. However, at temperatures greater than 100 °C, the  $\text{TiO}_2$  particles tended not to be adsorbed in some



**Fig. 2.** Scanning electron microscopic images of  $\text{TiO}_2$ /activated carbon particulates under various synthesis conditions: reaction temperatures were (a) 70 °C, (b) 80 °C, (c) 90 °C and (d) 120 °C under microwave heating conditions; using the oil-bath heating method the reaction temperatures were (e) 80 °C and (f) 90 °C; (g) naked activated carbon as the control.



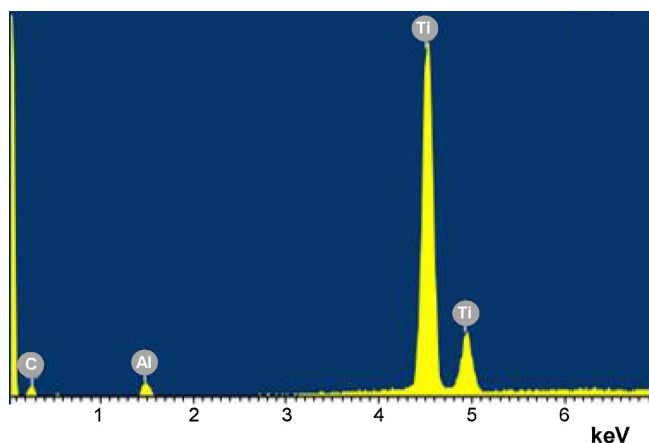


Fig. 3. SEM-EDX pattern of  $\text{TiO}_2$  coating on the activated carbon particulates upon heating to a temperature of  $70^\circ\text{C}$  by the microwave method.

sites of the AC surface. The results suggest that the optimal reaction temperature was  $90^\circ\text{C}$ .

The preparation of the composite  $\text{TiO}_2/\text{AC}$  particulates by the microwave heating method was compared to the more conventional oil-bath heating method. The SEM image of the resulting  $\text{TiO}_2/\text{AC}$  by the oil-bath method at  $80^\circ\text{C}$  is reported in Fig. 2e. Although the same heating rate and the same reaction temperature were used, the  $\text{TiO}_2$  formed on the AC surface was different from that of the microwave method (Fig. 2b) in that with oil-bath heating only a  $\text{TiO}_2$  thin film formed on the AC support at  $70^\circ\text{C}$ , and no changes occurred even when the temperature reached  $90^\circ\text{C}$  by the latter heating method (see Fig. 2f). Interestingly, similar findings were reported for the oil-bath heating method when the  $\text{TiO}_2$  precursor was titanium (IV) *iso*-propoxide solution [10]. No growth of  $\text{TiO}_2$  particles on activated carbon by the microwave method has hitherto been reported. Under our experimental conditions, no doubt the formation mechanisms of  $\text{TiO}_2$  on the AC surface differ between the microwave and the oil-bath heating methods as evident from the results observed by the SEM technique.

The morphology of free dispersed  $\text{TiO}_2$  particles formed in aqueous solution was also characterized by the SEM technique. At the reaction temperature at  $90^\circ\text{C}$ ,  $\text{TiO}_2$  particles of comparatively uniform particle size (ca. 460 nm) were observed by the microwave method (Fig. 5a). By contrast, using the oil-bath heating method to prepare  $\text{TiO}_2$  particles resulted in a rather non-uniform size distribution averaging around ca. 682 nm (Fig. 5b). Compared with  $\text{TiO}_2$  particles formed directly on the AC surface, the dispersed free  $\text{TiO}_2$  particles in solution were about 10% bigger making them unlikely

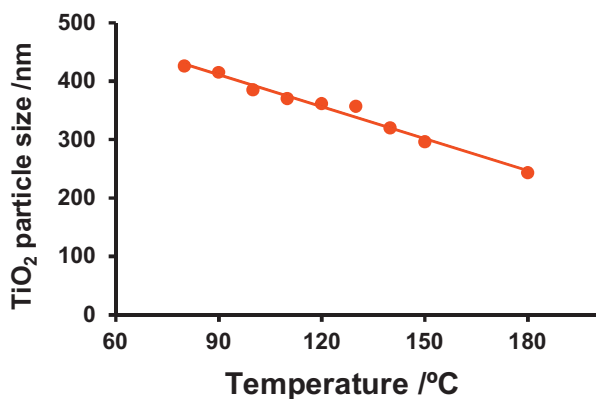


Fig. 4. Plot illustrating the decrease of  $\text{TiO}_2$  particle size on the AC surface at various reaction temperatures.

to be adsorbed on the AC surface. If such free  $\text{TiO}_2$  particles were adsorbed onto the AC surface, then we would expect the same results as obtained from particles formed when using the oil-bath method.

The UV–vis absorption spectra of naked AC particles and of the  $\text{TiO}_2/\text{AC}$  composites produced by the microwave method ( $90^\circ\text{C}$ ) are illustrated in Fig. 6a; the UV–vis absorption spectrum was also measured for the dispersed  $\text{TiO}_2$  particles in solution. The absorption of dispersed  $\text{TiO}_2$  particles in solution is clearly evident in the 350–400 nm range, from which we deduced that the band-gap energy of the  $\text{TiO}_2$  particles is ca. 3.3 eV by examining the expanded spectra in Fig. 6b, consistent with the anatase phase of the particles. By contrast, the absorption spectrum of  $\text{TiO}_2/\text{AC}$  particles seems not to be different from that of naked AC particles (Fig. 6a). The X-ray pattern of the  $\text{TiO}_2/\text{AC}$  composite particles produced by the microwave method ( $90^\circ\text{C}$ ) shown in Fig. 7 confirms the anatase nature of the  $\text{TiO}_2$  particulates.

The BET specific surface area of the synthesized  $\text{TiO}_2/\text{AC}$  composites was estimated to be about  $990\text{ m}^2\text{ g}^{-1}$  for the  $\text{TiO}_2/\text{AC}$  particles formed by the microwave method, while the specific surface area of the naked AC particles was somewhat smaller at  $922\text{ m}^2\text{ g}^{-1}$ . By contrast, the specific surface area of the  $\text{TiO}_2/\text{AC}$  particles prepared by the oil-bath method was about 14% lower at  $848\text{ m}^2\text{ g}^{-1}$ . Therefore, the  $\text{TiO}_2/\text{AC}$  particles synthesized by the microwave method should prove more advantageous for the adsorption of pollutants on the catalyst surface.

### 3.2. Proposed mechanism of the formation of $\text{TiO}_2/\text{AC}$ particles

A most important characteristic feature of heating a solvent medium by microwave irradiation is the dielectric loss ( $\epsilon''$ ) factor [11], which represents the quantity of input microwave energy lost to the sample by being dissipated as heat; it is a useful index of the generation of heat because of the interaction of the solvent with the microwave radiation field. The dielectric losses at a microwave frequency of 2.45 GHz were determined using an Agilent Technologies HP-85070B Network Analyzer and an Agilent dielectric high temperature probe (up to  $\sim 200^\circ\text{C}$ ) at various temperatures at  $5^\circ\text{C}$  intervals using a conventional plate heater; the volume of the sample was 100 mL in a Pyrex reactor. The temperatures of the solutions were measured with an optical fiber thermometer. The dielectric losses ( $\epsilon''$ ) of the AC particles dispersed in the aqueous titanium oxysulphate solution at various temperatures are collected in Fig. 8; those for pure water are also displayed for comparison to indicate that the general feature is a decrease of the dielectric loss with increase in temperature.

The initial dielectric loss of the dispersion ( $\epsilon'' = 33.4$ ) at near-ambient temperature decreased somewhat up to a temperature of  $60^\circ\text{C}$ , after which the dielectric loss tended to increase with temperature owing to some of microscopic chemical changes occurring in the  $\text{TiO}_2$  precursor titanium oxysulphate. This contrast can be explained by the efficient direct microwave heating of the activated carbon in the dispersion [12]. Accordingly, even though the AC particles are present in a high dielectric loss solvent such as water, selective heating of the AC particles nonetheless does occur under microwave irradiation [13] and thus provides the necessary energy for the formation and growth of the  $\text{TiO}_2$  particles.

The growth mechanism of  $\text{TiO}_2$  under microwave heating at  $70^\circ\text{C}$  and temperatures greater than  $80^\circ\text{C}$  conditions is proposed in the cartoons of Fig. 9. Both dispersed  $\text{TiO}_2$  particles in solution and a  $\text{TiO}_2$  thin coating on the AC surface formed by the efficient microwave heating method at  $70^\circ\text{C}$  (Fig. 9a) with the former  $\text{TiO}_2$  particles being dispersed in solution only. We suppose that the growth of the  $\text{TiO}_2$  thin coating took place through heat conduction from the solution. Under temperature conditions greater than  $80^\circ\text{C}$ , the heating efficiency of the water solvent by microwave

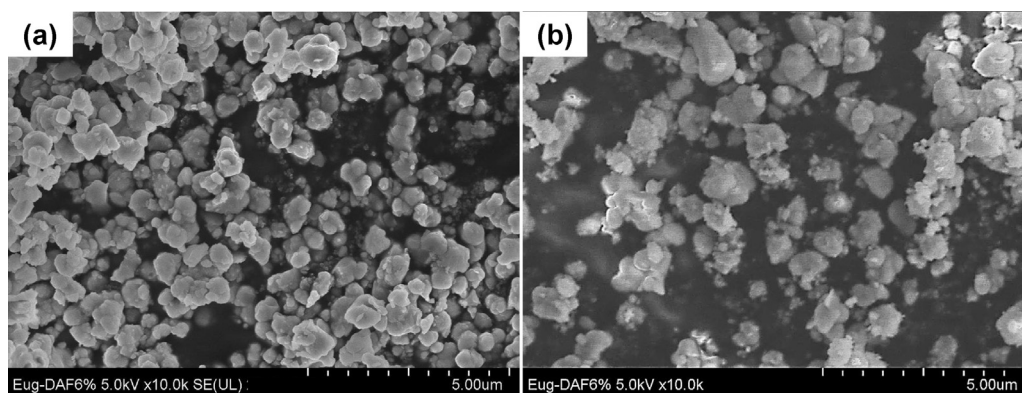


Fig. 5. Scanning electron microscopic images of free  $\text{TiO}_2$  particles in solution under heating at  $90^\circ\text{C}$  by (a) the microwaves and (b) by the oil-bath method.

heating decreases, whereas microwave direct heating of the AC particles increased (Fig. 9b). Evidently, direct heating of AC particles tended to produce  $\text{TiO}_2$  particles on the AC surface, together with smaller  $\text{TiO}_2$  particles formed at the higher reaction temperature.

In the case of the oil-bath heating method, formation of  $\text{TiO}_2$  on the AC surface only occurred through heat conduction from the heated oil outside the reactor, indicating that the growth mechanism did not depend on changes of temperature.

### 3.3. Photoactivity of MW-prepared $\text{TiO}_2/\text{AC}$ composite particles in the degradation of iso-propanol

Approximately 93–94% of iso-propanol (IPA) was either chemisorbed and/or physisorbed after 40 min under dark

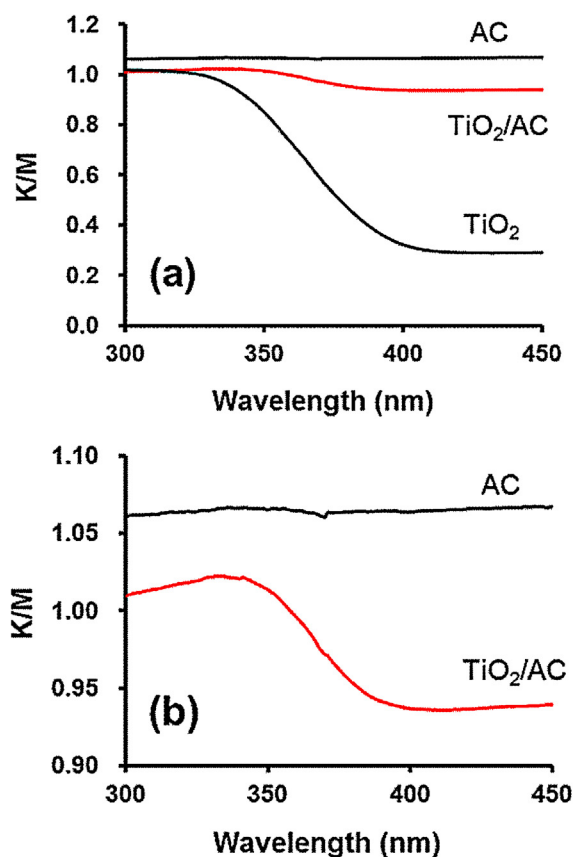


Fig. 6. (a) UV-vis absorption spectra of naked activated carbon (AC) particles and of the composite  $\text{TiO}_2/\text{AC}$  particles with  $\text{TiO}_2$  formed on activated carbon (AC) at a temperature of  $90^\circ\text{C}$ ; (b) expanded view of the absorption spectra of AC and  $\text{TiO}_2/\text{AC}$ .

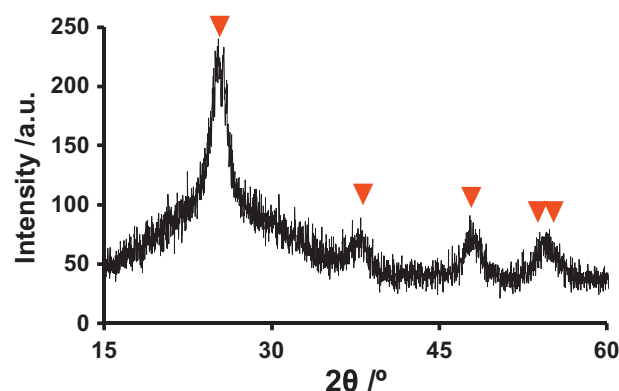


Fig. 7. X-ray diffraction pattern of the composite  $\text{TiO}_2/\text{AC}$  particles produced by the microwave method at the reaction temperature of  $90^\circ\text{C}$  (inverted triangles refer to the peak positions of the anatase crystalline form of  $\text{TiO}_2$ ).

conditions on the  $\text{TiO}_2/\text{AC}$  surface produced by microwave and oil-bath heating methods even though the specific surface areas differed. The extent of adsorption of IPA showed no further changes after 40 min, following which the  $\text{TiO}_2/\text{AC}/\text{IPA}$  particulates were irradiated with UV light thereby initiating the photodecomposition of the volatile organic compound IPA. The photodecomposition kinetics ( $C/C_0$  versus irradiation time) of iso-propanol with the microwave synthesized  $\text{TiO}_2/\text{AC}$  particles were  $12.4 \times 10^{-3} \text{ min}^{-1}$ , whereas with the  $\text{TiO}_2/\text{AC}$  particles produced by the oil-bath method the rate of degradation was nearly 6-fold slower at  $2.2 \times 10^{-3} \text{ min}^{-1}$  (see Fig. 10a). The degradation

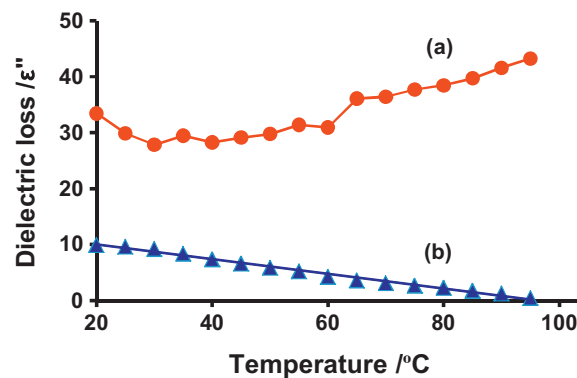
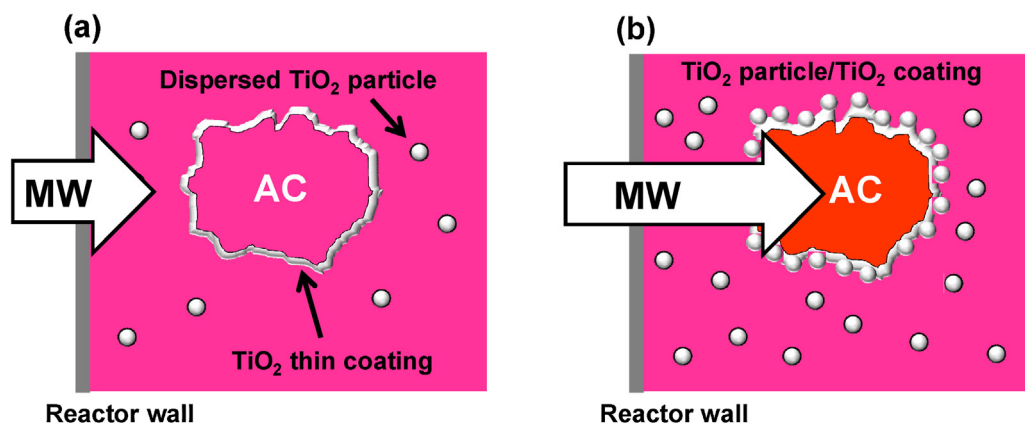
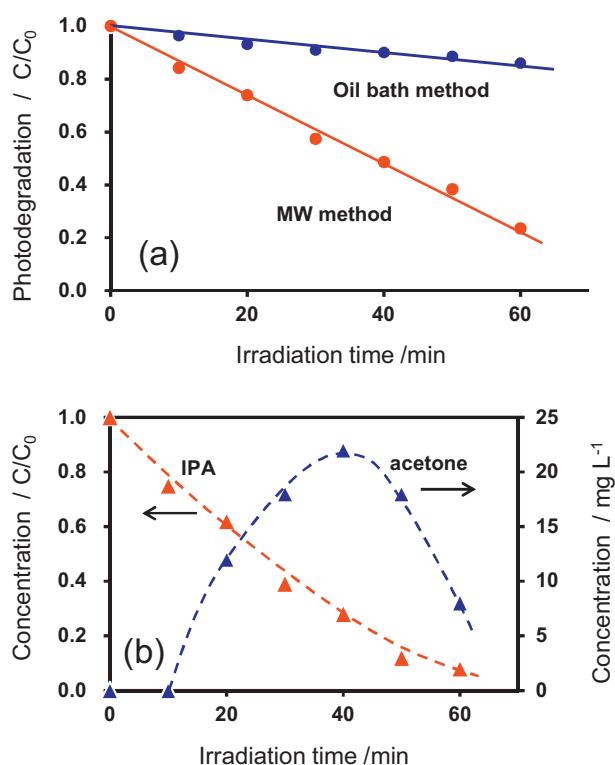


Fig. 8. Dielectric losses ( $\epsilon''$ ) at the microwaves frequency of 2.45 GHz for the (a) aqueous dispersion of activated carbon and titanium oxysulphate, and (b) pure water at various temperatures.



**Fig. 9.** Cartoon illustrating the growth mechanism of  $\text{TiO}_2/\text{AC}$  particles produced by the microwave heating method at a temperature of  $70^\circ\text{C}$  (a) and at temperatures greater than  $80^\circ\text{C}$  (b).



**Fig. 10.** (a) Photodecomposition kinetics of *iso*-propanol with the microwave- and oil bath- synthesized  $\text{TiO}_2/\text{AC}$  particles; (b) photodegradation of *iso*-propanol (IPA) and formation and degradation of the intermediate product acetone in the presence of  $\text{TiO}_2$  alone (no AC support).

efficiency was no doubt due to the formation of  $\text{TiO}_2$  particles on the AC surface.

It is well known that acetone and  $\text{CO}_2$  gas are produced as an intermediate and as the final product, respectively, by the photodecomposition of IPA. With the  $\text{TiO}_2/\text{AC}$  particles produced by the microwave method, the acetone intermediate formed after 20 min of UV irradiation; however, under these circumstances it was difficult to quantify the amount of acetone formed because it too could adsorb onto the  $\text{TiO}_2/\text{AC}$  particulates and undergo further degradation to carbon dioxide. A control experiment was conducted so as to confirm the photoactivity of  $\text{TiO}_2$  using  $\text{TiO}_2$  produced by the microwave method ( $90^\circ\text{C}$ ) in the absence of the AC support. Dried  $\text{TiO}_2$  particles (100 mg) were placed on a petri dish in

the reactor, after which  $300\text{ mg L}^{-1}$  (ppm) of IPA was added. After 20 min in the dark, approximately  $270\text{ mg L}^{-1}$  of IPA was chemically and/or physically adsorbed on the  $\text{TiO}_2$  surface; no further adsorption occurred after this time period. The photodecomposition of IPA and generation and degradation of the intermediate acetone are displayed in Fig. 10b. Recall that there is almost no difference in the initial adsorption under dark conditions between the particulates produced by the microwave method and the oil-bath method. Accordingly, we deduce that the photodegradation of IPA takes place through the mediation of the  $\text{TiO}_2$  particles formed on the AC surface.

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